Muon Liquid Handling System

User Guide

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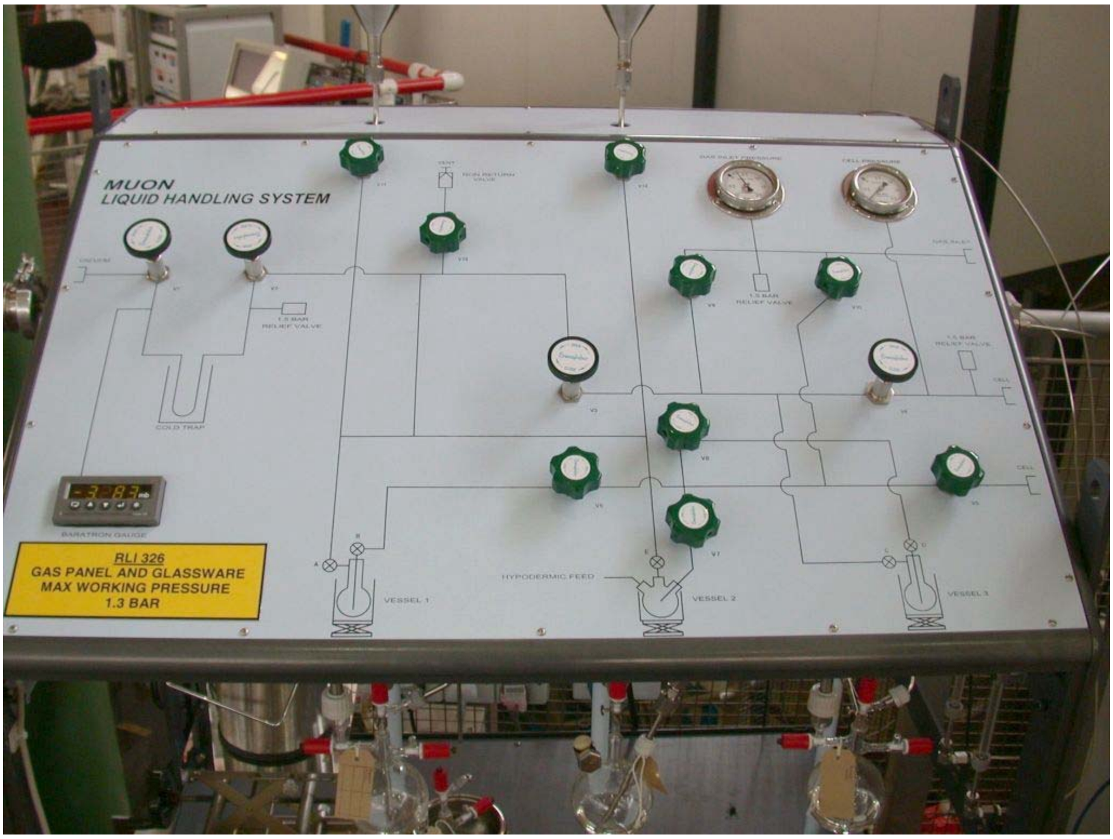
Version 0.2



Contents

1 Getting Started

This manual describes the liquid handling system and the *in situ* sample cell as used on the DEVA instrument with the "RF" spectrometer, for either normal muon spins relaxation or RF resonance experiments.

1.1 Layout of the liquid handling system

The Muon Liquid Handling System has been designed to facilitate the *in situ* degassing and transfer of samples into and out of the sample cell. The main features of the liquid handling system are shown in Figure 1.

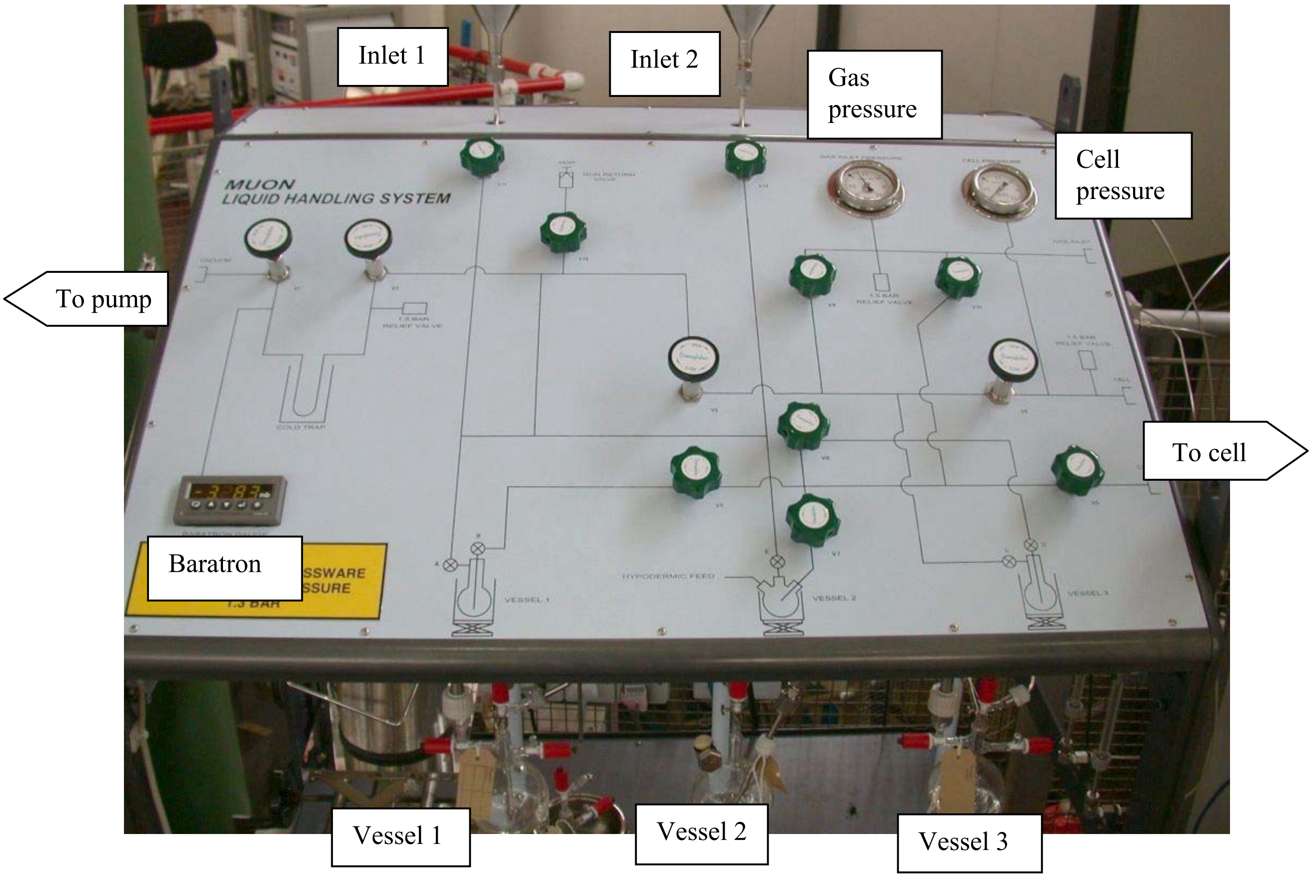


Figure 1 The layout of the front panel on the muon liquid handling system.

In general, the rig consists of three glass vessels for the storage of liquid samples, any of which can be opened to a vacuum for the purposes of degassing by the freeze-pump-thaw method. Once degassed, the sample liquid can be transferred to the sample cell contained within a cryostat without further exposure to the air. In addition, samples may be returned to the rig for further freeze-pump-thaw cycles or for disposal.

1.2 Layout of the *in situ* sample stick

The liquid sample stick is designed to fit into the DEVA flow cryostat, details of which can be found in the DEVA manual. It consists of a shapol target cell 30 mm x 30 mm with a mylar window upon which may be mounted an RF coil. Two stainless steel capillaries provide a means of flowing liquid into and out of the cell along with feed throughs for an RF excitation signal and a pick-up coil.

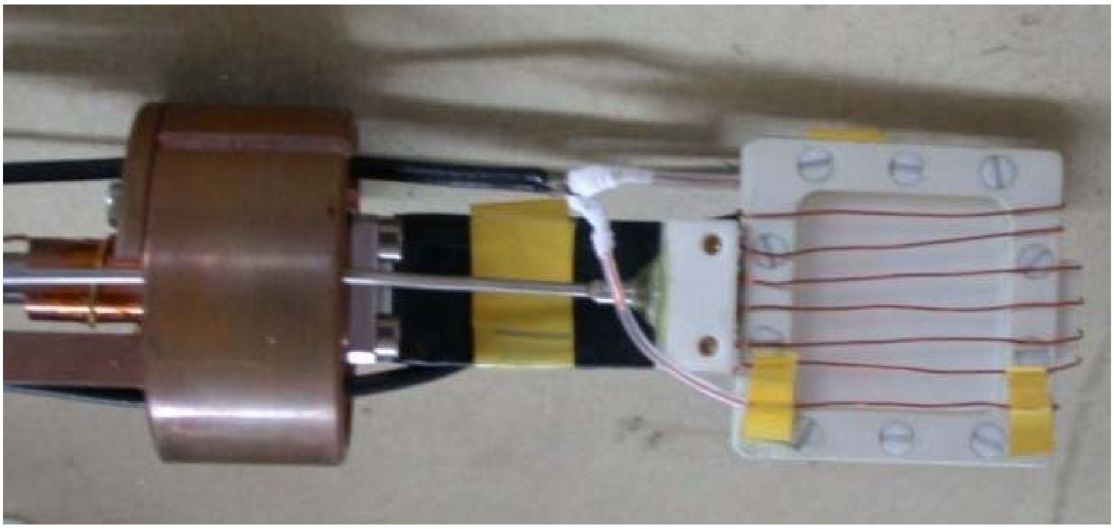


Figure 2 The liquid-sample cell mounted on a sample stick with a flat coil suitable for RF μSR.

1.3 Layout of the pump

The pump used with the Muon Liquid Handling System consists of a rotary pump used to reduce the pressure of large volumes of gas and a turbo pump to achieve a high vacuum. The main features pump used on the liquid handling system are shown in the diagram below.

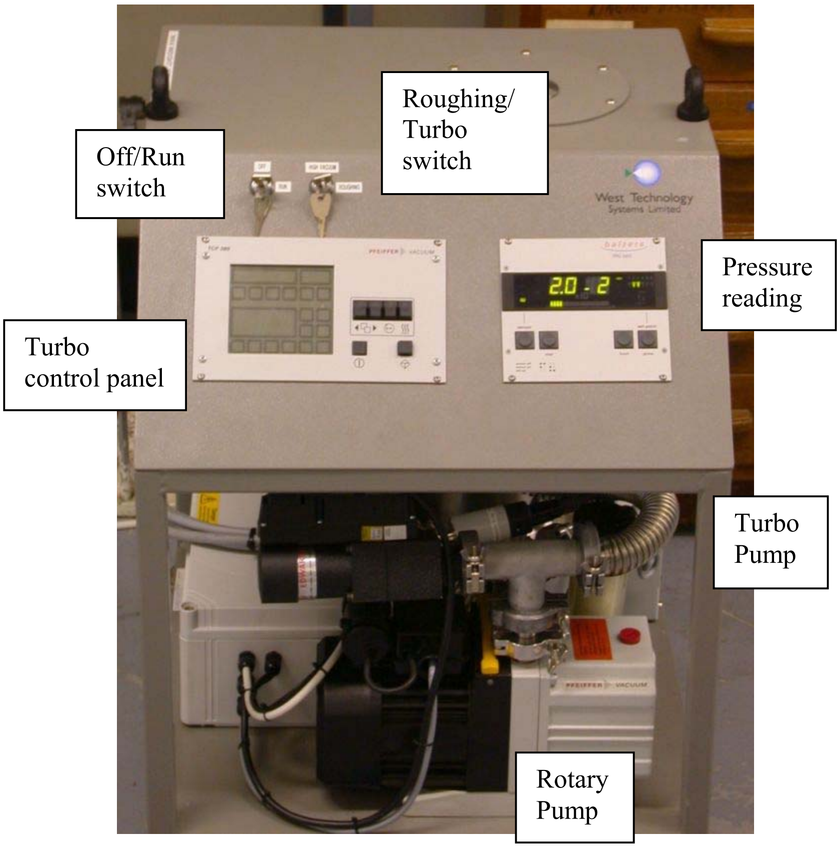


Figure 2 The layout of the front panel on the vacuum pump used with the muon liquid handling system.

2 General procedures

2.1 Sample loading

At this point, the system will be open to the atmosphere, ensure that valve V2 is closed to prevent air condensation in the cold trap.

2.1.1 Loading Vessel 1

- Open the tap on the right-hand side of Vessel 1.
- Open tap A and valve V11.
- The liquid should be poured into the system very slowly to avoid liquid leaving the vessel via the side tap.
- When loading is complete, close the side tap, tap A and valve V11.

2.1.2 Loading Vessel 2

- Ensure tap E and valve V7 are closed.
- Remove the rubber septum on Vessel 2 and open tap E.
- Opening valve 12 allows the liquid to be poured directly into the glass bulb.
- When loading is complete, close tap E and valve V12. Replace the rubber septum carefully making sure not to place any strain on the metal-to-glass seals.

It is important to note that a small amount of liquid may remain in the system after sample loading is finished. Care must be taken to prevent this liquid reaching the pump.

2.2 Evacuation of the system

After loading the sample liquid, the system must be evacuated. However, air and possibly a small amount of liquid will be in the main body of the system. To avoid air condensation in the cold trap, the nitrogen dewar surrounding it should be removed and the glass U-tube allowed to warm-up.

- Close all the taps on Vessels 1, 2 and 3.
- Ensure that the pump is switched to roughing before opening valve V1. The pump can be switched to turbo if a high vacuum is required.

Once the system is evacuated, air must be removed from the vessel containing the sample to be cleaned. The system must be 'let-up' to atmospheric pressure using helium gas. This is achieved by first closing valve V1 and then opening valves V3 and V9. The pressure of gas in the system can be read from the baratron gauge.

2.2.1 Removing air from Vessel 1

Once the system has been pressurised, tap A on Vessel 1 can be opened. It is possible to bubbled helium gas through the sample liquid against the non-return valve, V13. If this is required, first ensure that valves V5, V7 and V8 are closed. Open valves V10 and V6, finally opening tap B slowly in order to regulate the flow.

To fully evacuate the system and Vessel 1, the sample liquid must be frozen by raising a dewar of liquid nitrogen around it. When the sample is frozen, a vacuum can be achieved by switching the pump to its roughing mode and opening valve V1. For a high vacuum, switch the pump to turbo.

2.2.2 Removing air from Vessel 2

Once the system has been pressurised, tap E on Vessel 2 can be opened. It is possible to bubbled helium gas through the sample liquid against the non-return valve, V13. If this is required, first ensure that valves V5, V6 and V8 are closed. Valve V10 can now be opened and slowly opening V7 allows the gas flow to be regulated.

To fully evacuate the system and Vessel 2, the sample liquid must be frozen by raising a dewar of liquid nitrogen around it. When the sample is frozen, a vacuum can be achieved by switching the pump to its roughing mode and opening valve V1. For a high vacuum, switch the pump to turbo.

2.2.3 Removing air from Vessel 3

Once the system has been pressurised, tap C on Vessel 3 can be opened. It is possible to bubbled helium gas through the sample liquid against the non-return valve, V13. If this is required, first ensure that valves V5, V6 and V7 are closed. Valves V10 and V8 can now be opened and slowly opening tap D allows the gas flow to be regulated.

To fully evacuate the system and Vessel 3, the sample liquid must be frozen by raising a dewar of liquid nitrogen around it. When the sample is frozen, a vacuum can be achieved by switching the pump to its roughing mode and opening valve V1. For a high vacuum, switch the pump to turbo.

2.2.4 Evacuation of the sample cell

Once the system has been purged of oxygen, the sample cell can be evacuated. To do this, ensure that all the taps on the sample vessels are closed and the pump is switched to its roughing mode. Open valves V1, V2 and V3 to evacuate the system. Opening valve V4 will evacuate the sample cell. Switch the pump to turbo mode and leave to pump for as long as possible. When the sample cell is sufficiently clean, close valves V1 and V4 and let the system up to atmosphere using helium.

2.3 Freeze-Pump-Thaw cycle

Using this system it is possible to operate freeze-pump-thaw cycles on sample liquids held in any of the three vessels. Procedures appropriate to each vessel are outlined below.

2.3.1 Sample in Vessel 1

- All taps and valves should be closed.
- Open valve V2 and tap A on Vessel 1, and freeze the sample liquid by slowly raising a dewar of liquid nitrogen around it.
- When it has been determined that the liquid has completely frozen switch the pump to roughing mode and open valve V1.
- Switch the pump to turbo mode and leave for several minutes. The pressure within the system can be read from the baratron gauge on the pump.
- When sufficient pumping has been done and the sample is ready to thaw, close valve V1 and remove the dewar of liquid nitrogen from around Vessel 1.
- The system must be 'let-up' to atmospheric pressure using helium gas by opening valves V3 and V9. The pressure of gas in the system can be read from the baratron gauge.
- Tap B should be closed to prevent suck-back of the liquid as the solid thaws.
- The sample can be warmed gently to aid the thawing process and when complete close tap A.

2.3.2 Sample in Vessel 2

- All taps and valves should be closed.
- Open valve V2 and tap E on Vessel 2 and freeze the sample liquid by slowly raising a dewar of liquid nitrogen around it.
- When it has been determined that the liquid has completely frozen switch the pump to roughing mode and open valve V1.
- Switch the pump to turbo mode and leave for several minutes. The pressure within the system can be read from the baratron gauge on the pump.
- When sufficient pumping has been done and the sample is ready to thaw, close valve V1 and remove the dewar of liquid nitrogen from around Vessel 2.
- The system must be 'let-up' to atmospheric pressure using helium gas by opening valves V3 and V9. The pressure of gas in the system can be read from the baratron gauge.
- Valve V7 should be closed to prevent suck-back of the liquid as the solid thaws.
- The sample can be warmed gently to aid the thawing process and when complete close tap E.

2.3.3 Sample in Vessel 3

- All taps and valves should be closed.
- Open valves V2 and V3 along with tap C on Vessel 3 and freeze the sample liquid by slowly raising a dewar of liquid nitrogen around it.
- When it has been determined that the liquid has completely frozen switch the pump to roughing mode and open valve V1.
- Switch the pump to turbo mode and leave for several minutes. The pressure within the system can be read from the baratron gauge on the pump.
- When sufficient pumping has been done and the sample is ready to thaw, close valve V1 and remove the dewar of liquid nitrogen from around Vessel 3.
- The system must be 'let-up' to atmospheric pressure using helium gas by opening valves V3 and V9. The pressure of gas in the system can be read from the baratron gauge.
- Tap D should be closed to prevent suck-back of the liquid as the solid thaws.
- The sample can be warmed gently to aid the thawing process and when complete close tap C.

2.4 Liquid transfers

The transfer of liquids within the system is best accomplished by using relative differences in pressure brought about by cooling, evacuation or both.

2.4.1 Vessel 1 to sample cell

The transfer of liquid from Vessel 1 to the sample cell is brought about by the difference in pressure between that in the Vessel and a vacuum in the cell. However, in order to control the transfer of liquid the pressure difference should be as small as possible but not so low that the liquid starts to boil.

- Switch the pump to its roughing mode.
- Open value V2 and tap A, and carefully open valve V1 until a suitable pressure is obtained (~0.5 bar).
- Close tap A and continue to evacuate the system and sample cell by opening valves V3, V4, V5 and V6. Switch the pump to turbo mode.
- When the required level of vacuum has been obtained, close valves V4 and V5 in order to isolate the sample cell.
- Opening tap B on Vessel 1 will cause the liquid to move rapidly to fill the volume between tap B and V5.
- Valve V5 can be opened carefully in order to allow the liquid into the sample cell. The movement of liquid can be monitored using the sight glasses positioned either side of the sample cell.
- When it is judged that sufficient liquid has been transferred to fill the sample cell, close valve V5.

At this point, it must be remembered that the lower manifold contains liquid, which must be removed before the system is evacuated.

2.4.2 Vessel 3 to sample cell

The transfer of liquid from Vessel 3 to the sample cell is brought about by the difference in pressure between that in the Vessel and a vacuum in the cell. However, in order to control the transfer of liquid the pressure difference should be as small as possible but not so low that the liquid starts to boil.

- Switch the pump to its roughing mode.
- Open values V2 and V3 along with tap C and carefully open valve V1 until a suitable pressure is obtained (~0.5 bar).
- Close tap C and continue to evacuate the system and sample cell by opening valves V3, V4, V5 and V8. Switch the pump to turbo mode.
- When the required level of vacuum has been obtained, close valves V4 and V5 in order to isolate the sample cell.
- Opening tap D on Vessel 3 will cause the liquid to move rapidly to fill the volume between tap D and V5.
- Valve V5 can be opened carefully in order to allow the liquid into the sample cell. The movement of liquid can be monitored using the sight glasses positioned either side of the sample cell.
- When it is judged that sufficient liquid has been transferred to fill the sample cell, close valve V5.

At this point, it must be remembered that the lower manifold contains liquid, which must be removed before the system is evacuated.

2.4.3 Recovery of liquid in manifold to Vessel 2

After the transfer of liquid to or from the sample cell, some liquid will remain in the pipes of the lower manifold. This can be removed using the following procedure.

- Vessel 2 should be under vacuum prior to the loading of the liquid into the sample cell. If it is not, switch the pump to roughing mode and open tap E followed by valves V1 and 2. If Vessel 2 already contains some liquid ensure that it is frozen using liquid nitrogen before evacuation is attempted.
- When the vessel is evacuated close tap E and cool the glass bulb by raising a dewar of liquid nitrogen around it.
- Ensure that taps A, B, C, D and E are closed before opening valves V6 and V8.
- Opening valve V7 will allow any liquid present in the manifold to enter Vessel 2 where it will freeze.
- Close valve V7 and repressurise the system and Vessel 2 by opening valve V9 and tap E. Remove the dewar from around the vessel and allow the liquid to thaw.

2.4.4 Recovery of liquid in sample cell to Vessel 1

The liquid in the sample cell cannot be recovered to Vessel 1 using the liquid handling rig as presently configured.

2.4.5 Recovery of liquid in sample cell to Vessel 2

The liquid in the sample cell can be recovered to Vessel 2 using the following procedure.

- Vessel 2 should be under vacuum along with the manifold between valves V5 and V6 prior to the recovery of the liquid from the sample cell. If it is not, switch the pump to roughing mode and open tap E and valve V7 followed by valves V1 and V2. If Vessel 2 already contains some liquid, ensure that it is frozen using liquid nitrogen before evacuation is attempted.
- When the vessel and manifold have been evacuated, close tap E and cool the glass bulb by raising a dewar of liquid nitrogen around it.
- Ensure that tap E and valves V6 and V8 are closed before attempting to open valve V5.
- Opening valve V5 will allow the liquid present in the sample cell to be drawn into Vessel 2 where it will freeze.
- All the liquid may not return in a single pass and repeating the evacuation procedure may be required. The liquid may also be encouraged to leave the sample cell by using a small amount of gas.
- Close valve V3 and open valve V9 to allow helium gas into this part of the system. Close valve V9 and carefully open valve V4 to allow the gas to enter the sample cell. DO NOT repeat this procedure until the pressure in the system is known by opening tap E.
- Close valve V7 and re-pressurise the system and Vessel 2 by opening valve V9 and tap E.

2.4.6 Recovery of liquid in sample cell to Vessel 3

The liquid in the sample cell can be recovered to Vessel 3 using the following procedure.

- Vessel 3 should be under vacuum prior to the recovery of the liquid from the sample cell. If it is not, switch the pump to roughing mode and open tap C and valve V3 followed by valves V1 and V2. If Vessel 3 already contains some liquid, ensure that it is frozen using liquid nitrogen before evacuation is attempted.
- When the vessel and manifold have been evacuated, close valve V3 and cool the glass bulb by raising a dewar of liquid nitrogen around it.
- Ensure that tap D and valves V6, V7 and V8 are closed before attempting to open valve V4.
- Opening valve V4 will allow the liquid present in the sample cell to be drawn into Vessel 3 where it will freeze.

- All the liquid may not return in a single pass and repeating the evacuation procedure may be required. However, some liquid may be present in the system and care should be taken during evacuation that the trap does not become blocked. The liquid may also be encouraged to leave the sample cell by using a small amount of gas.
- Close valve V3 and open valve V10 to allow helium gas into the lower part of the system. Close valve V10 and carefully open valve V5 to allow the gas to pass through the sample cell. DO NOT repeat this procedure until the pressure in the system is known by opening valve V3.
- Close valve V4 and repressurise the system and Vessel 3 by opening valve V9.

3 Example Experiment

The paramagnetic signal in liquid n-hexane is difficult to observe because the muonium polarisation is found to decay on a microsecond time scale, which is further reduced by the presence of dissolved oxygen. A series of commissioning experiments were carried out using n-hexane in order to gauge the ability of the liquid handling rig to degas the liquid.

This experiment was carried out using a detachable liquid-sample cell.

At the start of the experiment, 100 ml of n-hexane was loaded into Vessel 1 and frozen to allow the removal of any dissolved gases. An inert atmosphere of helium gas was introduced and the liquid subjected to four freeze-pump-thaw cycles using the procedures outlined above. The sample cell was attached to one of the sample delivery tubes and pumped for 1 hr. Using the vacuum created in the cell, a small amount of n-hexane, enough to fill the cell, was transferred. To avoid contamination of the cleaned liquid, excess n-hexane was transferred to Vessel 3.

After filling, the sample cell was sealed and detached from the rig. Muon spin resonance experiments were performed at 2 G and 20 G followed by reattached to the rig. At this point, the rig was open to the air and so had to be evacuated and pumped for 30 minutes. The n-hexane held in the sample cell was transferred to Vessel 2 by freezing the vessel and allowing the vacuum to draw the n-hexane out of the cell. Gentle heating ensured that all the n-hexane was recovered.

Two further sets of freeze-pump-thaw cycles resulted in samples for muon study after 7 and 10 cycles had been performed on the clean n-hexane held in Vessel 1.

The signal due to the paramagnetic species in n-hexane after each set freeze-pump-thaw cycles is shown in Figure 1. The fitted parameters for the diamagnetic, paramagnetic and missing fraction percentage along with the relaxation rate for the paramagnetic signal are shown in Table 1.

Table 1 Effect of successive freeze-pump-thaw cycles on the initial asymmetry attributable to the diamagnetic, paramagnetic and missing fractions and muonium relaxation rate.

Experiment	Initial asymmetry (%)			Relaxation
	Diamagnetic	Paramagnetic	Missing	Rate (μs^{-1})
4 fpt cycles	12.87	3.41	6.72	7.96
7 fpt cycles	12.41	5.86	4.73	6.66
10 fpt cycles	12.86	4.90	5.24	5.45
6 fpt cycles sodium dried	12.59	4.39	6.02	5.20

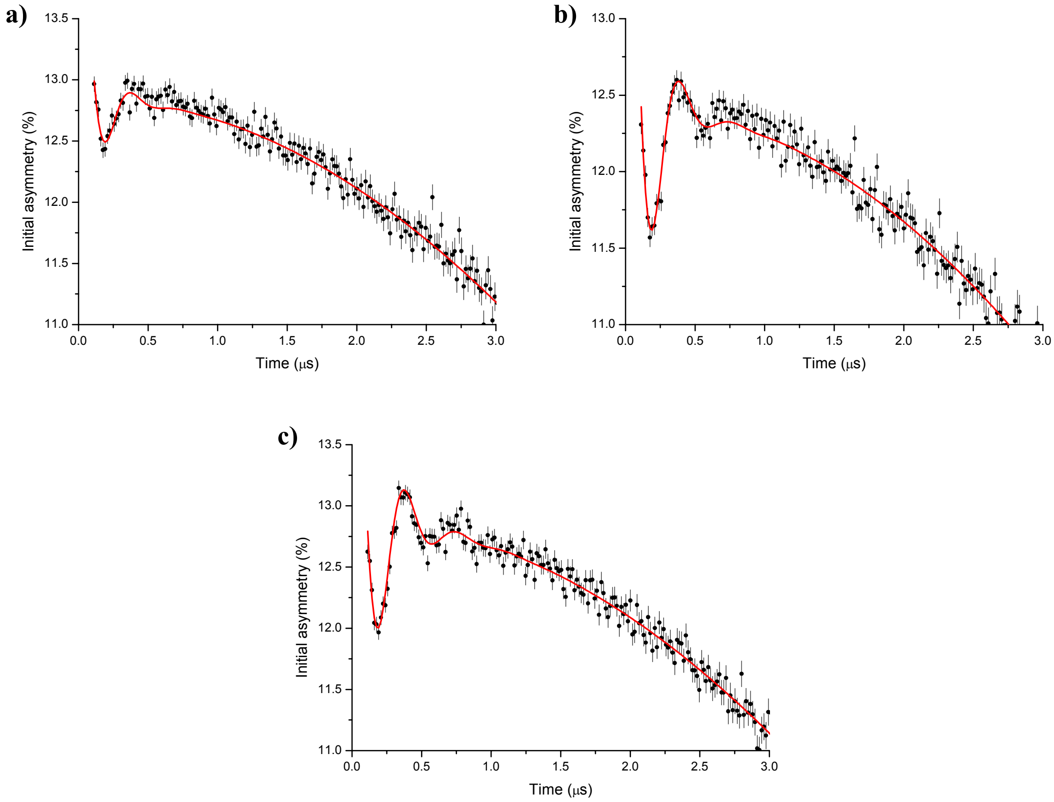


Figure 1 Muonium precession signal at 2G in n-hexane after a) 4, b) 7 and c) 10 freeze-pump-thaw cycles.

For the second experiment, all of the n-hexane used in the first experiment was recovered into a sample bottle and dried using a small piece of clean sodium. The liquid was carefully poured back into Vessel 1 and remounted on the rig. After freezing and subsequent evacuation and re-pressurisation with helium, the dry n-hexane was exposed to six freeze-pump-thaw cycles. Between each cycle, helium was bubbled through the liquid for several minutes. Again, the sample cell was attached to one of the sample delivery tubes and pumped for 1 hr before being loaded with n-hexane.